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Vitreous and porcelain enamels — Determination of resistance to chemical corrosion —

Part 3:
Determination of resistance to chemical corrosion by alkaline liquids using a hexagonal vessel

Émaux vitrifiés — Détermination de la résistance à la corrosion chimique —

Partie 3: Détermination de la résistance à la corrosion chimique par des liquides alcalins dans un récipient hexagonal

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 28706-3 was prepared by the European Committee for Standardization (CEN) (as EN 14483-3) and was adopted, under a special “fast-track procedure”, by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, in parallel with its approval by the ISO member bodies.

It cancels and replaces ISO 4533:1983 and ISO 4535:1983, which have been technically revised.

ISO 28706 consists of the following parts, under the general title *Vitreous and porcelain enamels — Determination of resistance to chemical corrosion*:

- *Part 1: Determination of resistance to chemical corrosion by acids at room temperature*
- *Part 2: Determination of resistance to chemical corrosion by boiling acids, boiling neutral liquids and/or their vapours*
- *Part 3: Determination of resistance to chemical corrosion by alkaline liquids using a hexagonal vessel*
- *Part 4: Determination of resistance to chemical corrosion by alkaline liquids using a cylindrical vessel*
- *Part 5: Determination of resistance to chemical corrosion in closed systems*

Introduction

Corrosion of vitreous and porcelain enamels by aqueous solutions is a dissolution process. The main component of the enamel, SiO_2, forms a three-dimensional silica network. After hydrolysis, it decomposes and forms silicic acid or silicates. These are released into the attacking medium. Other components, mainly metal oxides, are hydrolysed as well and form the corresponding hydrated metal ions or hydroxides. All corrosion products are more or less soluble in the attacking medium. The whole process results in a loss in mass per unit area.

For some aqueous solutions, the attack on the enamel proceeds linearly during the corrosion time; for other aqueous solutions, the attack on the enamel proceeds in a logarithmic manner during the corrosion time. Only for the first series of solutions can a scientifically exact rate of loss in mass per unit area ($g/m^2 \cdot h$) be calculated as well as a corrosion rate (mm/year).

The most important parameters influencing aqueous corrosion of the enamel are the enamel quality, the temperature and the pH-value. Inhibition effects resulting from the limited solubility of silica can also contribute. The following list describes different types of enamel attack for different corrosion conditions:

a) In aqueous alkali solutions like 0,1 mol/l NaOH (see Clause 9 of ISO 28706-4:2008), the silica network of the enamel is considerably attacked at 80 °C. Silicates and most of the other hydrolysed components are soluble in the alkali. Attack proceeds linearly during regular test times. Therefore, test results are expressed in terms of a rate of loss in mass per unit area (mass loss per unit area and time) and a corrosion rate (millimetres per year).

b) At room temperature, in weak aqueous acids like citric acid (see Clause 9 of ISO 28706-1:2008) or also in stronger acids like sulfuric acid (see Clause 10 of ISO 28706-1:2008), there is only minor attack on the silica network of the enamel. Other constituents are leached to some extent from the surface. Highly resistant enamels will show no visual change after exposure. On less resistant enamels, some staining or surface roughening will occur.

c) In boiling aqueous acids (see ISO 28706-2), the silica network of the enamel is being attacked, and silica as well as the other enamel components are released into solution. However, the solubility of silica in acids is low. Soon, the attacking solutions will become saturated with dissolved silica and will then only leach the surface. The acid attack is inhibited and the rate of corrosion drops markedly.

   NOTE The glass test equipment also releases silica by acid attack and contributes to the inhibition of the corrosion.

   Inhibition is effectively prevented in vapour phase tests. The condensate formed on the test specimen is free of any dissolved enamel constituents.

   Examples of enamel corrosion proceeding in a logarithmic manner [see 1)] and linearly [see 2)] are:

   1) **Boiling citric acid (see Clause 10 of ISO 28706-2:2008) and boiling 30 % sulfuric acid (see Clause 11 of ISO 28706-2:2008)**

      Since only minute amounts of these acids are found in their vapours, the test is restricted to the liquid phase. The attack is influenced by inhibition effects, and corrosion depends on the time of exposure. Therefore, test results are expressed in terms of loss in mass per unit area; no rate of loss in mass per unit area is calculated.

   2) **Boiling 20 % hydrochloric acid (see Clause 12 of ISO 28706-2:2008)**

      Since this is an azeotropic boiling acid, its concentration in the liquid and the vapour phase are identical, and liquid phase testing need not be performed. Vigorous boiling supplies an uninhibited condensate, and the attack proceeds linearly with time of exposure. Therefore, test results are only expressed in terms of rate of loss in mass per unit area (mass loss per unit area and time) and the corrosion rate (millimetres per year).

d) At high temperatures, with tests in the liquid phase under autoclave conditions (see ISO 28706-5), aqueous acid attack is severe. To avoid inhibition, the test time is restricted to 24 h and the ratio of attacking acid to attacked enamel surface is chosen so that it is comparatively high (similar to that in a chemical reaction vessel). In addition, only low-silica water is used for the preparation of test solutions. Under these conditions, attack will proceed linearly with time of exposure. Therefore, test results with 20 % hydrochloric acid (see Clause 8 of ISO 28706-5:2008), artificial test solutions (see Clause 10 of ISO 28706-5:2008) or process fluids (see Clause 11 of ISO 28706-5:2008) are also expressed in terms of a rate of loss in mass per unit area (loss in mass per unit area and time).

e) In boiling water (see Clause 13 of ISO 28706-2:2008), the silica network is fairly stable. The enamel surface is leached and silica is dissolved only to a small extent. This type of attack is clearly represented by the vapour phase attack. In the liquid phase, some inhibition can be observed with highly resistant enamels. However, if the enamel being tested is weak, leached alkali from the enamel can raise pH-values to alkaline levels, thus increasing the attack by the liquid phase. Both liquid and vapour phase testing can give valuable information.

f) Since the attack may or may not be linear, the results are expressed only in terms of loss in mass per unit area, and the test time should be indicated.

g) For standard detergent solution (see Clause 9 of ISO 28706-3:2008), it will not be certain whether the linear part of the corrosion curve will be reached during testing for 24 h or 168 h. Calculation of the corrosion rate is therefore not included in the test report.

h) For other acids (see Clause 14 of ISO 28706-2:2008) and other alkaline solutions (see Clause 10 of ISO 28706-3:2008 and Clause 10 of ISO 28706-4:2008), it will also not be known if a linear corrosion rate will be reached during the test period. Calculation of the corrosion rate is therefore not included in the test reports of those parts of this International Standard.

For vitreous enamels fired at temperatures below 700 °C, the test parameters (media, temperatures and times) of this International Standard are not appropriate. For such enamels, for example aluminium enamels, other media, temperatures and/or times should be used. This can be done following the procedures described in the clauses for “Other test solutions” in Parts 1, 2, 3 and 4 of this International Standard.

Vitreous and porcelain enamels — Determination of resistance to chemical corrosion —

Part 3:
Determination of resistance to chemical corrosion by alkaline liquids using a hexagonal vessel

1 Scope

This part of ISO 28706 describes a test method for the determination of the resistance of vitreous and porcelain enamelled articles to attack by alkaline liquids at temperatures between 25 °C and 95 °C. The apparatus used is a hexagonal vessel in which six enamelled specimens are simultaneously tested.

NOTE 1 The resistance to any alkaline liquid can be determined. However, the test method was originally used for the determination of the resistance to hot detergent solutions, within the neutral and alkaline range, used for washing textiles.

NOTE 2 Since detergents are continually subject to alterations in their composition, a standard test solution is specified which, in respect to its alkalinity, wetting properties and complexing behaviour, can be considered as a typical composition for the detergents at present on the market. The pH-value and alkalinity of the standard test solution depend on the proportions of sodium tripolyphosphate, sodium carbonate and sodium perborate present; sodium tripolyphosphate acts simultaneously as a complexing agent. The wetting properties of the standard test solution are obtained by the addition of alkylsulfonate. A higher sodium perborate content is not considered necessary since the effect of oxygen on enamel is unimportant and an increase in the perborate content does not cause any significant alteration in the alkalinity of the standard test solution. The testing of different enamels using this standard test solution and other test solutions (including 5 % sodium pyrophosphate solution) has justified the use of this standard test solution for determining the resistance of enamels to hot detergent solutions.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 48, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 28764, *Vitreous and porcelain enamels — Production of specimens for testing enamels on sheet steel, sheet aluminium and cast iron*

3 Principle

Six similarly enamelled specimens are simultaneously exposed to attack by an alkaline liquid under specified conditions of temperature and time, the solution being continuously stirred during the test.

The loss in mass is determined and used to calculate the rate of loss in mass per unit area.

NOTE In order to correspond to the conditions of a washing machine used in practice, the alkaline liquid is stirred during the test. The solution is cold when put into the vessel and is heated to the desired temperature in the vessel.

4 Reagents

During the determination, use only reagents of recognized analytical grade, unless otherwise specified.

4.1 Water, conforming to the requirements of grade 3 of ISO 3696, i.e. distilled water or water of equivalent purity.

4.2 Degreasing solvent, such as ethanol, or water (4.1) containing a few drops of liquid detergent, suitable for cleaning the test apparatus and test specimens.

4.3 Sodium tripolyphosphate ($Na_5P_3O_{10}$).

4.4 Sodium carbonate (Na_2CO_3), anhydrous.

4.5 Sodium perborate, hydrated ($NaBO_2 \cdot H_2O_2 \cdot 3H_2O$).

4.6 Sodium silicate, containing about 81 % (by mass) of Na_2SiO_3.

4.7 Alkylsulfonate $[CH_3(CH_2)_x - C(SO_2Na)H - (CH_2)_3 - CH_3]$.

5 Apparatus and materials

5.1 Test apparatus

5.1.1 General description

The apparatus (see Figures 1 to 4) consists of a hexagonal vessel having a circular opening in each side. A specimen is pressed against each of these openings by means of gripping plates which are held in place by wing nuts, sealing rings being placed between the vessel and the specimens. A lid having four holes, for a paddle stirrer, two immersion heaters and a temperature-controlling device, is screwed on to the vessel, a sealing ring being placed between the vessel and the lid. The paddle stirrer, immersion heaters and temperature-controlling device are fixed such that their distance from the bottom of the vessel is 30 mm.

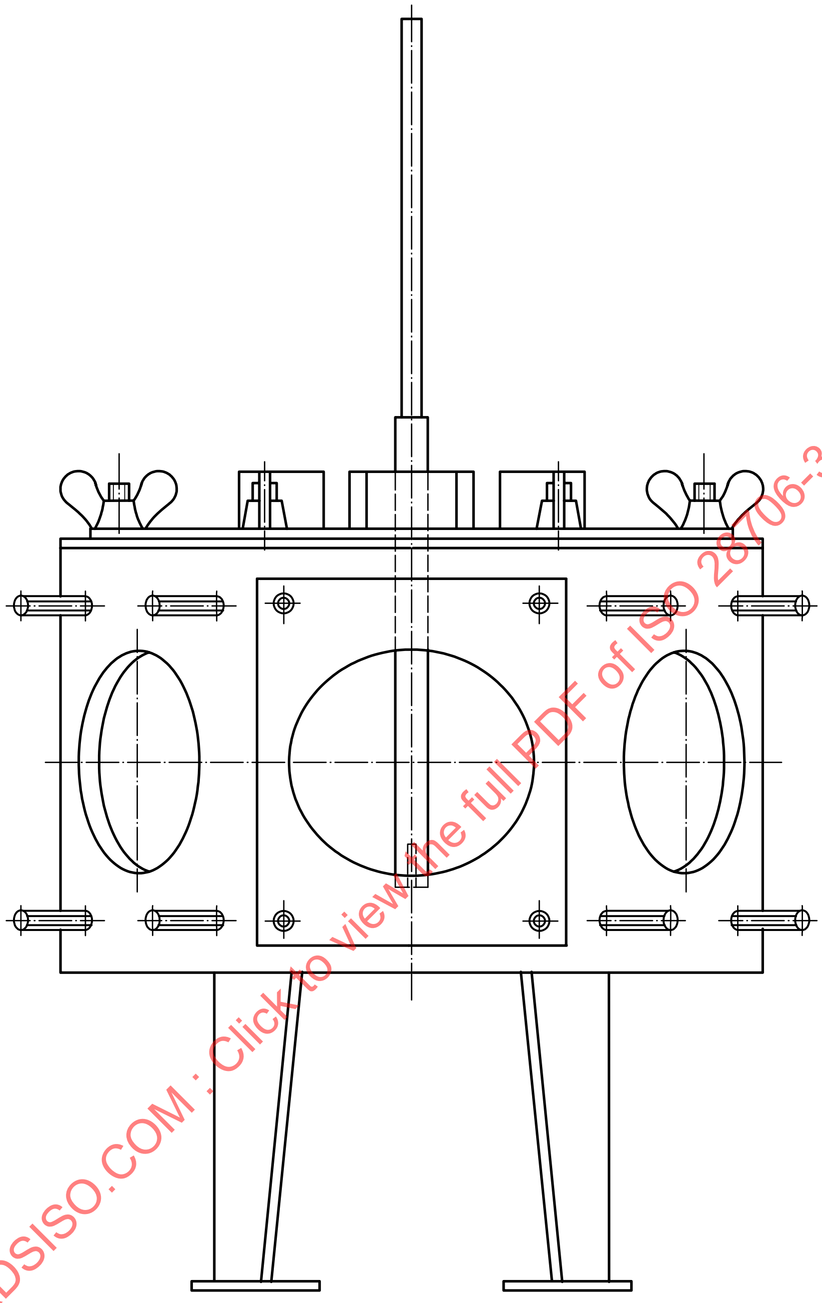
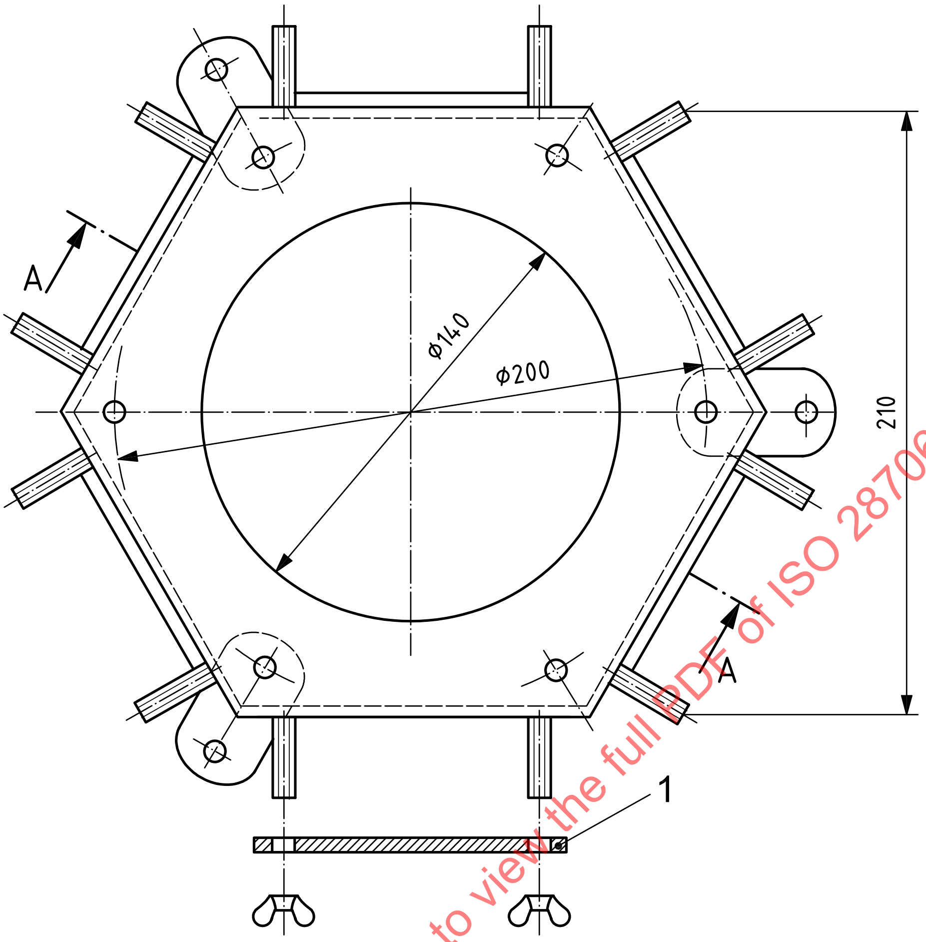


Figure 1 — Hexagonal vessel with lid, stirrer and gripping plate

Dimensions in millimetres

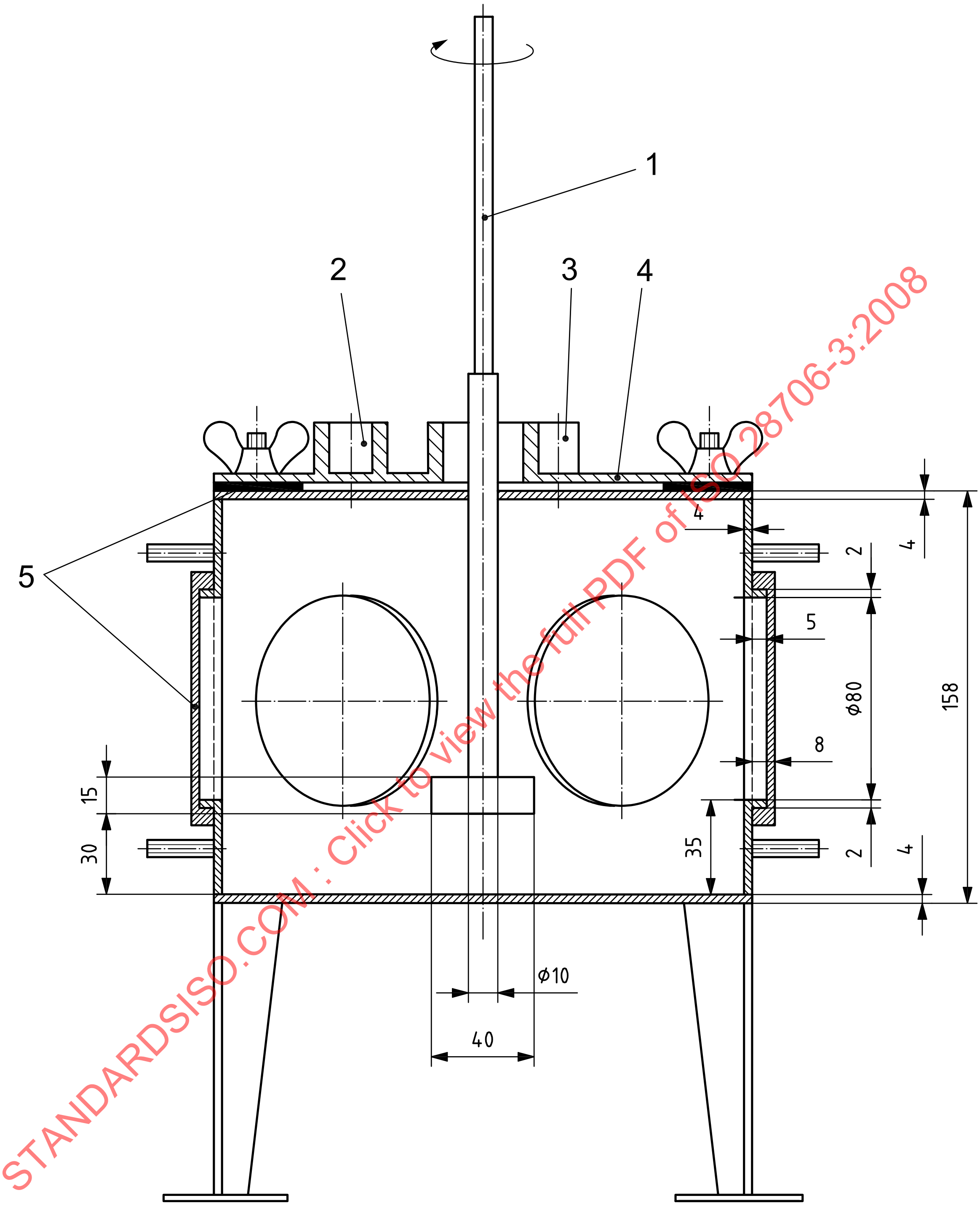


Key

1 gripping plate

Figure 2 — Top view of hexagonal vessel without lid and paddle stirrer

Dimensions in millimetres



Key

1 paddle stirrer
2 socket for temperature-controlling device
3 socket for immersion heater
4 lid
5 sealing rings

Figure 3 — Section A-A of the hexagonal vessel, lid and paddle stirrer, showing sealing rings

Dimensions in millimetres

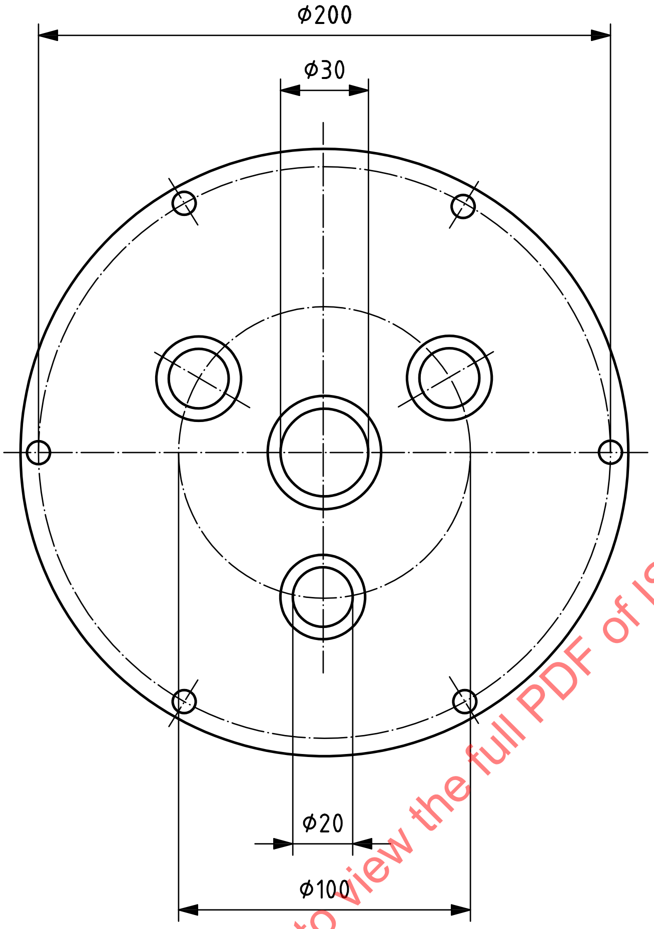


Figure 4 — Top view of lid

The sealing rings (5.1.6) shall be made of synthetic rubber of hardness 70 IRHD as determined in accordance with ISO 48. The material shall be resistant to alkaline solutions at 100 °C (chloroprene, for example, is suitable).

The hexagonal vessel, lid, gripping plates and paddle stirrer shall be made of the same austenitic stainless steel.

5.1.2 Hexagonal vessel, of austenitic stainless steel (see Figures 1 to 3), with four threaded bolts welded to each side for fastening the gripping plates and with six threaded bolts welded to the upper surface for fastening the lid. The vessel should preferably have an outlet for drainage.

5.1.3 Lid, of austenitic stainless steel, with a centrally placed support for receiving the paddle stirrer and with three further supports for receiving the immersion heaters and the temperature-controlling device.

5.1.4 Gripping plates (six), of austenitic stainless steel of thickness 4 mm, which can be fitted to the sides of the hexagonal vessel.

5.1.5 Wing nuts (30), for fastening the gripping plates and the lid to the vessel.

5.1.6 Sealing rings (six), of external diameter 100 mm, internal diameter 80 mm and thickness 8 mm, for sealing the side openings. An additional ring, of internal diameter 140 mm and of thickness 3 mm, is required to serve as an intermediate layer between the lid and the vessel.

5.1.7 Paddle stirrer, of austenitic stainless steel, with the dimensions shown in Figure 3. The stirrer shall operate at a rotational frequency of (1 350 ± 50) min^{-1}.

5.1.8 Immersion heaters (two), cylindrical, each of 600 W, made of nickel-plated copper or of austenitic stainless steel.

5.1.9 Temperature-controlling device, comprising a contact thermometer with a thermostat accurate to ± 1 °C. The use of a temperature-recording instrument is recommended.

5.2 Hot-air oven, capable of being maintained at (120 ± 5) °C.

5.3 Desiccator, for example with an internal diameter of 200 mm.

5.4 Balance, accurate to 0,2 mg.

5.5 Cotton wool.

6 Test specimens

The specimens to be used shall be prepared in accordance with ISO 28764. Enamel both sides of the specimens.

7 Procedure

For each determination, two tests with two similarly enamelled specimens shall be carried out.

Before the test, wipe each specimen with cotton wool (5.5) soaked in the degreasing solvent (4.2). Then dry the specimens for 2 h in the hot-air oven (5.2), controlled at (120 ± 5) °C, cool for at least 2 h in the desiccator (5.3) and weigh to the nearest 0,2 mg. Record the starting mass, m_s.

Press the specimens against the side openings of the hexagonal vessel and secure them by means of the gripping plates so that the vessel is watertight. Pour 4,5 l of the alkaline test solution (see Clauses 9 and 10), at room temperature, into the vessel through the inlet in the lid. Heat the test solution to the specified temperature (see Clauses 9 and 10), stirring continuously, and maintain it at this temperature for the specified time (see Clauses 9 and 10).

At the end of the required time (see Clauses 9 and 10), remove the hot test solution and fill the vessel immediately with water (4.1) at room temperature. Stir the water (4.1) for 2 min and then remove it. Remove the specimens from the vessel and rinse the vessel thoroughly once more.

Wipe both sides of the specimens with cotton wool soaked in water (4.1) and then rinse with the degreasing solvent (4.2). Dry the specimens for 2 h in the hot-air oven (5.2), controlled at (120 ± 5) °C, and then leave them in the desiccator (5.3) for 2 h. Weigh each specimen to the nearest 0,2 mg and record the final mass, m_f.

Measure the diameter of the area exposed to attack. The mean value of three measurements of the diameter of the area exposed to attack shall lie within ± 1 mm of 80 mm. Calculate the area exposed to attack, A, using this mean value of the diameter.

8 Expression of results

For each test, calculate the result as the total loss in mass per unit area, $\Delta\rho_\text{A}$, in g/m², for the total duration of the test, using the equation:

$$\Delta\rho_\text{A} = \frac{(m_\text{s} - m_\text{f})}{A} \qquad (1)$$

where

m_s is the starting mass, in g;

m_f is the final mass, in g;

A is the area exposed to attack, in m^2.

In order to distinguish between the test results for different test periods, the number of test hours shall be stated as a subscript to the symbol; for example, for a test period of 24 h, $\Delta\rho_{A24}$.

Results for test specimens which show defects such as pinholes down to the metal, chipped edges or edge corrosion shall be discarded and a corresponding number of new specimens shall be tested.

Express the result as the arithmetic mean of the individual values to the nearest 0,1 g/m^2. The individual values shall not differ from the mean value by more than 20 %.

9 Standard detergent solution test

9.1 General

Carry out this test following the procedure described in Clause 7.

9.2 Test solution

Prepare 4,5 l of a solution containing the following:

- 27,0 g of sodium tripolyphosphate ($Na_5P_3O_{10}$);
- 9,0 g of anhydrous sodium carbonate (Na_2CO_3);
- 2,7 g of hydrated sodium perborate ($NaBO_2 \cdot H_2O_2 \cdot 3H_2O$);
- 1,8 g of sodium silicate, containing about 81 % (by mass) of Na_2SiO_3;
- 4,5 g of alkylsulfonate [$CH_3(CH_2)_x - C(SO_2Na)\,H - (CH_2)_3 - CH_3$].

The solution shall be made up using water (4.1) and reagents of analytical grade.

Prepare a fresh test solution for each test.

NOTE Each 24 h of test uses 4,5 l of the test solution.

9.3 Test temperature

The test solution shall be heated in the vessel to 95 °C and shall be maintained at that temperature for the duration of the test.

9.4 Duration of the test

The heating time at 95 °C (i.e. without the heating-up time) shall be 24 h.

If the average loss in mass per unit area is less than 8 mg after 24 h, repeat the test with new sets of specimens, increasing the test period to 168 h. Replace the standard test solution after each 24 h period by removing the hot test solution and filling the vessel immediately with fresh test solution at room temperature.